reference method analytical procedure is out of control. Corrective action must be taken to determine the source of the error(s) (e.g., calibration standard discrepancies, extraction problems, etc.) and the reference method and audit sample determinations must be repeated according to paragraph (c) of this section, or the entire test procedure (starting with paragraph (a) of this section) must be repeated.

(h) *Test for comparability.* (1) For each filter pair, calculate all nine possible percent differences (D) between the reference and candidate methods, using all nine possible combinations of the three determinations (A, B, and C) for each method, as:

Equation 6

$$D_{in} = \frac{C_{ij} - R_{ik}}{R_{ik}} \times 100\%$$

where:

i is the filter number, and n numbers from 1 to 9 for the nine possible difference combinations for the three determinations for each method (j = A, B, C, candidate; k = A, B, C, reference).

(2) If none of the percent differences (D) exceeds ±20 percent, the candidate method passes the test for comparability.

(3) If one or more of the percent differences (D) exceeds ±20 percent, the candidate method fails the test for comparability.

(i) The candidate method must pass both the precision test (paragraph (f) of this section) and the comparability test (paragraph (h) of this section) to qualify for designation as an equivalent method.

§**53.34   Test procedure for methods for PM$_{10}$ and PM$_{2.5}$.**

(a) *Collocated measurements.* Set up three reference method samplers collocated with three candidate method samplers or analyzers at each of the number of test sites specified in table C–4 of this subpart. At each site, obtain as many sets of simultaneous PM$_{10}$ or PM$_{2.5}$ measurements as necessary (see paragraph (c)(3) of this section), each set consisting of three reference method and three candidate method measurements, all obtained simultaneously.

For PM$_{2.5}$ candidate Class II equivalent methods, at least two collocated PM$_{10}$ reference method samplers are also required to obtain PM$_{2.5}$/PM$_{10}$ ratios for each sample set. Candidate PM$_{10}$ method measurements shall be 24-hour integrated measurements; PM$_{2.5}$ measurements may be either 24- or 48-hour integrated measurements. All collocated measurements in a sample set must cover the same 24- or 48-hour time period. For samplers, retrieve the samples promptly after sample collection and analyze each sample according to the reference method or candidate method, as appropriate, and determine the PM$_{10}$ or PM$_{2.5}$ concentration in µg/m³. If the conditions of §53.30(d)(4) apply, collect sample sets only with the three reference method samplers. Guidance for quality assurance procedures for PM$_{2.5}$ methods is found in section 2.12 of the Quality Assurance Handbook (reference 6 of appendix A to subpart A of this part).

(b) *Sequential samplers.* For sequential samplers, the sampler shall be configured for the maximum number of sequential samples and shall be set for automatic collection of all samples sequentially such that the test samples are collected equally, to the extent possible, among all available sequential channels or utilizing the full available sequential capability.

(c) *Test for comparability and precision.* (1) For each of the measurement sets, calculate the average PM$_{10}$ or PM$_{2.5}$ concentration obtained with the reference method samplers:

Equation 7

$$\overline{R}_j = \frac{\sum_{i=1}^{3} R_{ij}}{3}$$

where:

R denotes results from the reference method;
i is the sampler number; and
j is the set.

(2)(i) For each of the measurement sets, calculate the precision of the reference method PM$_{10}$ or PM$_{2.5}$ measurements as:

Equation 8

$$P_j = \sqrt{\dfrac{\sum\limits_{i=1}^{3} R_{ij}^2 - \dfrac{1}{3}\left(\sum\limits_{i=1}^{3} R_{ij}\right)^2}{2}}$$

If the corresponding R_j is below:

80 $\mu g/m^3$ for PM_{10} methods.
40 $\mu g/m^3$ for 24-hour $PM_{2.5}$ at single test sites for Class I candidate methods.
40 $\mu g/m^3$ for 24-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios >0.75.
30 $\mu g/m^3$ for 48-hour $PM_{2.5}$ at single test sites for Class I candidate methods.
30 $\mu g/m^3$ for 48-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios >0.75.
30 $\mu g/m^3$ for 24-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios <0.40.
20 $\mu g/m^3$ for 48-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios >0.75.

(ii) Otherwise, calculate the precision of the reference method PM_{10} or $PM_{2.5}$ measurements as:

Equation 9

$$Rp_j = \dfrac{1}{\overline{R}_{-j}} \sqrt{\dfrac{\sum\limits_{i=1}^{3} R_{ij}^2 - \dfrac{1}{3}\left(\sum\limits_{i=1}^{3} R_{ij}\right)^2}{2}} \times 100\%$$

(3) If R_j falls outside the acceptable concentration range specified in table C–4 of this subpart for any set, or if P_j RP_j as applicable, exceeds the value specified in table C–4 of this subpart for any set, that set of measurements shall be discarded. For each site, table C–4 of this subpart specifies the minimum number of sample sets required for various conditions, and § 53.30(b)(5) specifies the $PM_{2.5}/PM_{10}$ ratio requirements applicable to Class II candidate equivalent methods. Additional measurement sets shall be collected and analyzed, as necessary, to provide a minimum of 10 acceptable measurement sets for each test site. If more than 10 measurement sets are collected that meet the above criteria, all such measurement sets shall be used to demonstrate comparability.

(4) For each of the acceptable measurement sets, calculate the average PM_{10} or $PM_{2.5}$ concentration obtained with the candidate method samplers:

Equation 10

$$\overline{C}_j = \dfrac{\sum\limits_{i=1}^{3} C_{ij}}{3}$$

where:
C denotes results from the candidate method;
i is the sampler number; and
j is the set.

(5) For each site, plot the average PM_{10} or $PM_{2.5}$ measurements obtained with the candidate method (R_j) against the corresponding average PM_{10} or $PM_{2.5}$ measurements obtained with the reference method (R_j). For each site, calculate and record the linear regression slope and intercept, and the correlation coefficient.

(6) If the linear regression parameters calculated under paragraph (c)(5) of this section meet the values specified in table C–4 of this subpart for all test sites, the candidate method passes the test for comparability.

[62 FR 38792, July 19, 1997; 63 FR 7714, Feb. 17, 1998]

TABLE C–1 TO SUBPART C—TEST CONCENTRATION RANGES, NUMBER OF MEASUREMENTS REQUIRED, AND MAXIMUM DISCREPANCY SPECIFICATION

Pollutant	Concentration Range Parts per Million	Simultaneous Measurements Required				Maximum Discrepancy Specification, Parts per Million
		1–hr		24–hr		
		First Set	Second Set	First Set	Second Set	
Ozone	Low 0.06 to 0.10	5	6	....	....	0.02
	Med 0.15 to 0.25	5	6	....	....	.03
	High 0.35 to 0.45	4	6	....	....	.04
	Total	14	18			
Carbon Monoxide	Low 7 to 11	5	6	....	....	1.5
	Med 20 to 30	5	6	....	....	2.0

TABLE C–1 TO SUBPART C—TEST CONCENTRATION RANGES, NUMBER OF MEASUREMENTS REQUIRED, AND MAXIMUM DISCREPANCY SPECIFICATION—Continued

Pollutant	Concentration Range Parts per Million	Simultaneous Measurements Required				Maximum Discrepancy Specification, Parts per Million
		1–hr		24–hr		
		First Set	Second Set	First Set	Second Set	
	High 35 to 45	4	6	....	....	3.0
	Total	14	18			
Sulfur Dioxide	Low 0.02 to 0.05	....	....	3	3	0.02
	Med 0.10 to 0.15	....	....	2	3	.03
	High 0.30 to 0.50	7	8	2	2	.04
	Total	7	8	7	8	
Nitrogen Dioxide	Low 0.02 to 0.08	....	....	3	3	0.02
	Med 0.10 to 0.20	....	....	2	3	.03
	High 0.25 to 0.35	....	....	2	2	.03
	Total	....	....	7	8	

TABLE C–2 TO SUBPART C—SEQUENCE OF TEST MEASUREMENTS

Measurement	Concentration Range	
	First Set	Second Set
1	Low	Medium
2	High	High
3	Medium	Low
4	High	High
5	Low	Medium
6	Medium	Low
7	Low	Medium
8	Medium	Low
9	High	High
10	Medium	Low
11	High	Medium
12	Low	High
13	Medium	Medium
14	Low	High
15	....	Low
16	....	Medium
17	....	Low
18	....	High

TABLE C–3 TO SUBPART C—TEST SPECIFICATIONS FOR LEAD METHODS

Concentration range, $\mu g/m^3$	0.5–4.0
Minimum number of 24-hr measurements	5
Maximum analytical precision, percent	5
Maximum analytical accuracy, percent	±5
Maximum difference, percent of reference method	±20

TABLE C–4 TO SUBPART C—TEST SPECIFICATIONS FOR PM_{10} AND $PM_{2.5}$ METHODS

Specification	PM_{10}	$PM_{2.5}$	
		Class I	Class II
Acceptable concentration range (R_j), $\mu g/m^3$	30–300	10–200	10–200
Minimum number of test sites	2	1	2
Number of candidate method samplers per site	3	3	3
Number of reference method samplers per site	3	3	3
Minimum number of acceptable sample sets per site for PM_{10}:			
$R_j < 80$ $\mu g/m^3$	3		
$R_j > 80$ $\mu g/m^3$	3		
Total	10		
Minimum number of acceptable sample sets per site for $PM_{2.5}$:			

TABLE C–4 TO SUBPART C—TEST SPECIFICATIONS FOR PM$_{10}$ AND PM$_{2.5}$ METHODS—Continued

Specification	PM$_{10}$	PM$_{2.5}$	
		Class I	Class II
Single test site for Class I candidate-equivalent methods:			
$\quad$ R$_j$ < 40 μg/m^3 for 24-hr or R$_j$ < 30 μg/m^3 for 48-hr samples		3	
$\quad$ R$_j$ > 40 μg/m^3 for 24-hr or R$_j$ > 30 μg/m^3 for 48-hr samples		3	
Sites at which the PM$_{2.5}$/PM$_{10}$ ratio must be > 0.75:			
$\quad$ R$_j$ < 40 μg/m^3 for 24-hr or R$_j$ < 30 μg/m^3 for 48-hr samples			3
$\quad$ R$_j$ > 40 μg/m^3 for 24-hr or R$_j$ > 30 μg/m^3 for 48-hr samples			3
Sites at which the PM$_{2.5}$/PM$_{10}$ ratio must be < 0.40:			
$\quad$ R$_j$ < 30 μg/m^3 for 24-hr or R$_j$ < 20 μg/m^3 for 48-hr samples			3
$\quad$ R$_j$ > 30 μg/m^3 for 24-hr or R$_j$ > 20 μg/m^3 for 48-hr samples			3
Total, each site		10	10
Precision of replicate reference method measurements, P$_j$ or RP$_j$ respectively, maximum	5 μg/m^3 or 7%	2 μg/m^3 or 5%	2 μg/m^3 or 5%
Slope of regression relationship	1±0.1	1±0.05	1±0.05
Intercept of regression relationship, μg/m^3	0±5	0±1	0±1
Correlation of reference method and candidate method measurements	≥0.97	≥0.97	≥0.97

[62 FR 38792, July 18, 1997; 63 FR 7714, Feb. 17, 1998]

FIGURE C–1 TO SUBPART C—SUGGESTED FORMAT FOR REPORTING TEST RESULTS

Candidate Method☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐

Reference Method☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐

Applicant☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐

☐ First Set ☐ Second Set ☐ Type ☐ 1 Hour ☐ 24 Hour

Concentration Range		Date	Time	Concentration, ppm		Difference	Table C–1 Spec.	Pass or Fail
				Candidate	Reference			
Low ☐☐☐☐ ppm to ☐☐☐☐ ppm1	1							
	2							
	3							
	4							
	5							
	6							
Medium ☐☐☐☐ ppm to ☐☐☐☐ ppm1	1							
	2							
	3							
	4							
	5							
	6							
High ☐☐☐☐ ppm to ☐☐☐☐ ppm1	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
							Total Failures:	

APPENDIX A TO SUBPART C—
REFERENCES

(1) American National Standard—Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, ANSI/ASQC E4-1994. Available from American Society for Quality Control, 611 East Wisconsin Avenue, Milwaukee, WI 53202.

Subpart D—Procedures for Testing Performance Characteristics of Methods for PM$_{10}$

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